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Effect of Processing Methods and Fiber Content on Mechanical and Abrasive Wear Performance of Sisal Fiber/PLA Biocomposites

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Abstract

In this work, an attempt is made to study the effect of processing methods and fiber content on mechanical properties and abrasive wear behavior of polylactide-based biocomposites reinforced with sisal fiber. Three processing methods namely film-stacking, melt-impregnation and solvent-impregnation were used for fabricating unidirectional sisal fiber-reinforced biocomposites. The impact of processing methods and fiber content on biocomposites was assessed through evaluation of mechanical properties, viscoelastic behavior and abrasive wear performance. Morphology was studied by scanning electron microscopy. Biocomposites processed by solvent-impregnation method exhibited enhancement of tensile and flexural properties and reduction in Izod impact strength. Experimental results of abrasive wear tests revealed the influence of processing methods and fiber content on wear resistance of biocomposites.

Keywords: *biocomposite, solvent impregnation, mechanical properties, abrasive wear*

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INTRODUCTION

The depletion of petroleum resources, plastic disposal problems, and emissions during incineration along with increasing environmental regulations have led to increased interest in development of green composite materials that are compatible with the environment and independent of fossil fuels [1, 2]. These new-generation ecofriendly materials developed with renewable resource-based biopolymers and natural fibers, can compete in markets currently dominated by products based on petroleum feedstock in applications such as packaging, automotive and consumer goods. However, high moisture-absorption capabilities, poor fiber-matrix adhesion, and improper processing methods are the key factors that are limiting their range of applications.

During the past two decades, extensive research was carried out on improving the mechanical properties of the biocomposites via physical and chemical treatments [3–6]. However, much attention has not been paid on assessing the impact of processing methods on mechanical properties and abrasive wear performance of biocomposites. Lui *et al.* [7] studied the influence of injection and compression molding on mechanical and thermal properties of biocomposites from kenaf and soy-based bioplastic. They found that compression-molded specimens exhibited higher heat deflection temperature and notched Izod impact strength. Franco and Gonzalez [8] found that pre-impregnation of henequen fiber in HDPE/xylene solution, produced a significant improvement in tensile strength. Mohanty *et al.* [9] adopted powder impregnation process for fabricating biocomposites from chopped natural fiber and polypropylene powder. In another work, Mohanty *et al.* [10] used powder impregnation through compression molding (process-I) and extrusion followed by injection molding (process-II) for fabrication of hemp fiber and cellulose acetate biocomposites. They experienced that process-II produced superior

strength biocomposites over their counterparts fabricated by using process-I. Gomes *et al.* [11] developed two new methods, namely, preforming and prepreg sheet method for fabrication of unidirectional curaua fiber green composites. They found that prepreg sheet composite has superior tensile strength, compared to the composite fabricated with performing method. Garkhail *et al.* [12] demonstrated that mechanical properties of flax fibers-reinforced PHB composites are greater when processed using film stacking (hot press forming) in comparison to injection molding. Chand and Dwivedi [13] found that addition of MA-g-PP coupling agent during melt mixing gives better wear resistance as compared to jute PP composite having MA-g-PP solution-treated jute fibers.

Reviewing the research carried out by various researchers, it is understood that processing method can be considered as the key factor in determining the performance of the biocomposites. There is very little information in the literature relating to the influence of processing methods on mechanical and wear performance of biocomposites. Hence, the main focus of this work was to evaluate the impact of three different processing methods and fiber content, on mechanical and wear performance of the unidirectional sisal fiber-reinforced biocomposites.

MATERIALS AND METHODS

Materials

Biodegradable polymer, polylactide (PLA) (GCS PLA4320) supplied by Green Chemical Co. Ltd., South Korea, was used as a matrix in this work. Physical, mechanical and thermal properties of the virgin matrix are given in Table 1. Sisal fiber with a density of 1.29 g/cm^3 and an average diameter of 60 µm was used for reinforcing polylactide. Mean tensile strength obtained from single fiber tests was found to be 432 ± 41 N/mm^2.

Table 1: *Physical and Mechanical Properties of Polylactide.*

Density (g/cm^3)	1.25
Melt flow index (g/10 min)(210 °C/2.16 kg)	15
Tensile strength (MPa)	42
Tensile modulus (GPa)	1.7
Flexural strength(MPa)	85
Melting temperature (°C)	170
Glass transition temperature (°C)	65

Composite Fabrication

Three different processing methods are employed for the fabrication of sisal fiber-reinforced composites, namely, film stacking, melt impregnation and solvent impregnation to evaluate the effect of processing methods on mechanical properties and abrasive wear performance.

Film-Stacking Method (FS)

In this method, PLA films were prepared by heating and pressing PLA granules at 170 °C. Film thickness was maintained constant by using a picture frame of 0.7 mm thick. Subsequently, PLA films and unidirectional stitched sisal fiber mats were stacked alternately in a flash picture frame mold (180 × 180 × 3 mm^3) and pressed between the hot plates of hydraulic hot press at a temperature and pressure of 170 °C and 5 MPa respectively for 5 min.

Melt-Impregnation Method (MI)

Composites were fabricated by hot pressing of prepregs produced via melt-impregnation method. Initially, prepregs were prepared by placing unidirectional stitched mat of controlled weight in between PLA films in a metallic mold and pressed slightly for 60 min between the hot plates of hydraulic hot press maintained at 170 °C. Finally, melt-impregnated prepregs were stacked in a flash picture frame mold and pressed at temperature and pressure of 170 °C and 5 MPa respectively for 5 min.

Solvent-Impregnation Method (SI)

In this method, prepregs were prepared by impregnation of sisal fiber in PLA/chloroform solution. Initially, controlled weight of PLA was dissolved in chloroform and poured onto unidirectional stitched sisal fiber mat placed in a stainless steel tray and solvent was allowed

to evaporate slowly at room temperature. Eventually, prepregs obtained were subsequently dried in vacuum at 60 °C for 24 h to remove the traces of chloroform. Finally, composites were fabricated by hot pressing the solvent-impregnated prepregs in a picture frame mold by following the same conditions as used in melt-impregnation method.

Mechanical Tests

Tensile and three-point flexural tests were conducted using a universal testing machine (INSTRON 3385). Tensile and flexural tests were carried out in accordance with ASTM D 638 (Type I) and ASTM D 790 respectively. Cross-head speeds of 5 mm/min and 2 mm/min were used for tensile and flexural tests respectively. Notched Izod impact strength was measured using Tinius Olsen impact tester according to ASTM D 256. All the tests were replicated ten times, and the mean value was reported.

Scanning Electron Microscopy

Tensile fractured surfaces were examined by a Zeisis EVO MA scanning electron microscope (SEM) with an acceleration voltage of 10 kV. Prior to SEM observations, samples were sputter coated with gold to make them conductive.

Dynamic Mechanical Analysis

Storage modulus, loss modulus and loss factor (tan δ) of the composite specimens (60 mm × 12 mm × 3 mm) were measured as a function of temperature (25–120 °C) using Q800 DMA (TA instruments) in the dual cantilever bending mode. Tests were carried out at a constant heating rate of 5 °C/min and frequency of 1 Hz.

Abrasive Wear Tests

Abrasive wear tests were performed using pin-on-disc apparatus (Ducom make) to study the effect of processing method on wear performance of the biocomposites. Specimens for abrasive wear tests having dimensions of 3 mm × 3 mm × 25 mm were cut from the compression-molded plates. Specimens were abraded against a rotating disc, on which abrasive paper of 600 grit size was mounted using double-sided adhesive tape. The tests were conducted in 100, 200, 300, 400, 500, 600, cycles corresponding to sliding distances of 25.12 m, 50.24 m, 75.36 m, 100.48 m, 125.60 m, 150.72 m, respectively. A constant sliding speed of 0.418 m/s and a constant load of 9.8 N were applied. Weight loss at the end of each set of 100 cycles was measured using Shimadzu high precision balance.

RESULTS AND DISCUSSION

Mechanical Properties

Tensile strength and moduli of unidirectional sisal fiber-reinforced biocomposites are shown in Figures 1 and 2 respectively. Influence of processing methods and fiber content on the tensile properties of biocomposites is manifest from these figures. Among the three processing methods studied in this work, composites fabricated with solvent-impregnation method yielded higher tensile properties, whereas the film-stacking method resulted in lower tensile strength and modulus. In the solvent impregnation method, low viscosity of PLA/chloroform solution would enable better flow of resin through the fiber, and thus result in better impregnation and wetting of the fiber in the matrix, which also helps in improving fiber matrix adhesion. In the case of film-stacking technique, molten matrix has to penetrate through the fiber without displacing them. However, high melt viscosity of the matrix restricts its flow through the fiber, resulting in insufficient wetting of fiber in the matrix. Melt viscosity increases drastically at higher temperatures, which allow better wettability of fiber in the matrix, but these higher processing temperatures are intentionally avoided preventing thermal degradation of sisal fiber. Thus, higher tensile properties observed in solvent-impregnated composites may be due to better wettability and proper impregnation of fiber in the matrix when compared to film-stacking method. Unlike the expectations, melt-impregnation method resulted in lower tensile properties than that of the solvent-impregnation method. This result is inconsistent with the work reported by Gomes *et al.* [11], which states that melt-impregnated curaua fiber composites had a higher tensile strength than solvent-impregnated composites. This discrepancy may be attributed to the low temperature

resistance of natural fiber [14], resulting in thermal degradation, when exposed to a temperature of 170°C for 1 h. Ochi [15] observed 20% strength reduction due to thermal degradation, in hemp fiber exposed to temperature of 180°C for one1 h.

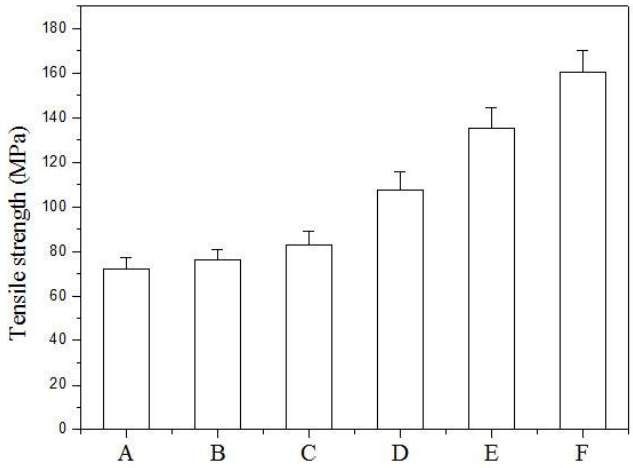

Fig. 1: *Tensile Strength of Sisal Fiber-Reinforced Biocomposites for (A) 20 wt% Sisal Fiber FS Composite (B) 20 wt% Sisal Fiber MI Composite (C) 20 wt% Sisal Fiber SI Composite (D) 30 wt% Sisal Fiber SI Composite (E) 40 wt% Sisal Fiber SI Composite (F) 50 wt% Sisal Fiber SI Composite.*

SEM images of tensile fractured surfaces of film-stacked and solvent-impregnated composites were shown in Figure 3. The fractured surface of film-stacked composite exhibited more pull outs, which is an indication of poor wettability and fiber matrix adhesion, whereas micrograph of solvent-impregnated composite exhibited a fiber breakage rather than pull outs. This indicates the stress transfer between fiber and matrix pursuant to better wettability.

Modified rule of mixtures for unidirectional fiber-reinforced composites given in Eq. (1) was used to evaluate the reinforcement efficiency factor:

$$\sigma_c = \xi_c(V_f \sigma_f) + (1-Vf)\sigma_m \quad (1)$$

where, σ_c, σ_f, σ_m are tensile strengths of composite, fiber and matrix respectively. V_f is the volume fraction of the fiber and ξ_c is fiber reinforcement efficiency factor, which depends on fiber length, aspect ratio and fiber matrix adhesion. Weight fractions of sisal fiber are converted into volume fractions by Eq. (2).

$$Vf = \frac{\frac{Wf}{\rho f}}{\frac{Wf}{\rho f}+\frac{Wm}{\rho m}} \quad (2)$$

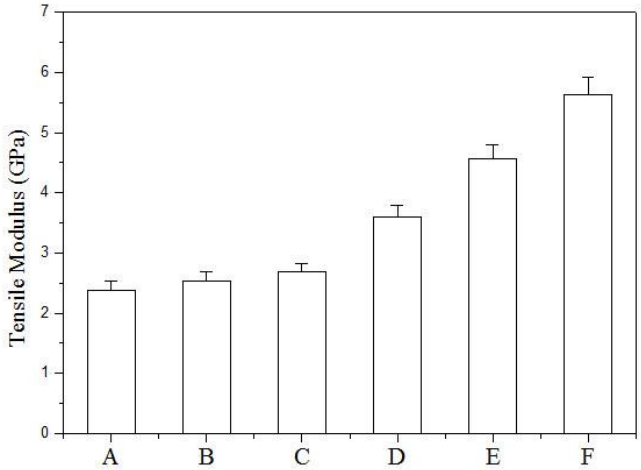

Fig. 2: *Tensile Modulus of Sisal Fiber-Reinforced Biocomposites for (A) 20 wt% Sisal Fiber FS Composite (B) 20 wt% Sisal Fiber MI Composite (C) 20 wt% Sisal Fiber SI Composite (D) 30 wt% Sisal Fiber SI Composite (E) 40 wt% Sisal Fiber SI Composite (F) 50 wt% Sisal Fiber SI Composite.*

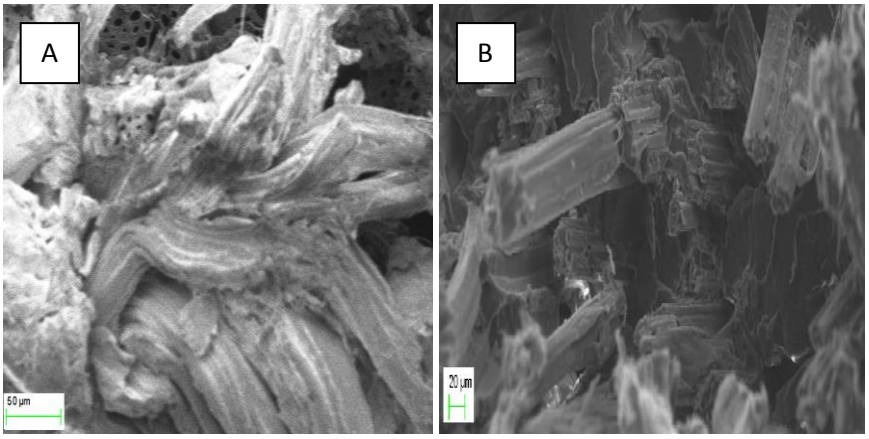

Fig. 3: *SEM Images of Tensile Fractured Surfaces of (A) Film-Stacked Composite (B) Solvent-Impregnated Composite.*

where, W_f and W_m are the weight fractions of the fiber and matrix respectively. ρ_f and ρ_m are the densities of fiber and matrix respectively. Reinforcement efficiency factors for composites fabricated with film-stacking, melt-impregnation and solvent-impregnation methods are found to be 0.51, 0.56 and 0.59 respectively. This clearly indicates the profound influence of processing methods on reinforcement efficiency.

Tensile strength and modulus increased with progressive increments of fiber content and resulted in maximum around fiber content of 50% by weight. At higher fiber contents (i.e., at 60%), some voids were observed, which may be due to insufficient wetting of fiber in the matrix. Hence, it is concluded that 50 wt% fiber content is the optimum percentage for fabricating composite with sisal fiber having an average diameter of 60 µm.

Flexural strength and modulus of sisal fiber-reinforced unidirectional composites are shown in Figures 4 and 5 respectively. It can be observed that both flexural strength and modulus are significantly affected by processing method and fiber content. It is further observed that composite fabricated with solvent-impregnation method has higher flexural strength and modulus. In flexure mode, failure occurs due to separation between fibers and matrix [15], hence higher flexural properties of solvent-impregnated composites indicates better adhesion between fiber and matrix.

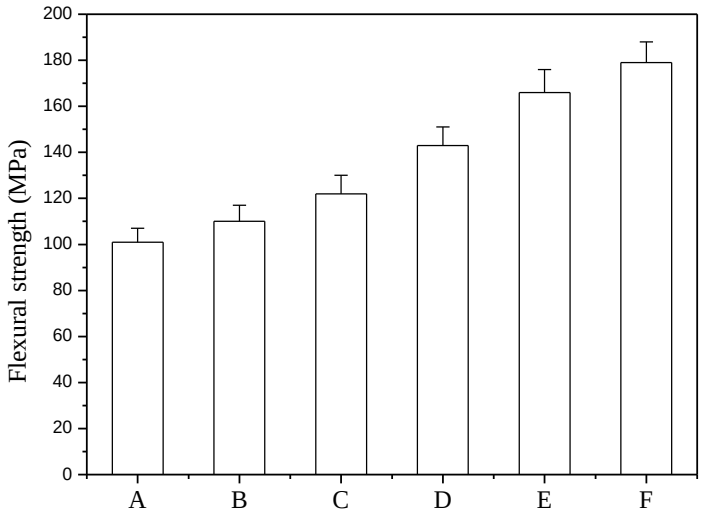

Fig. 4: *Flexural Strength of Sisal Fiber-Reinforced Biocomposites for (A) 20 wt% Sisal Fiber FS Composite (B) 20 wt% Sisal Fiber MI Composite (C) 20 wt% Sisal Fiber SI Composite (D) 30wt% Sisal Fiber SI Composite (E) 40 wt% Sisal Fiber SI Composite (F) 50 wt% Sisal Fiber SI Composite.*

Effect of processing methods and fiber content on notched Izod impact strength of unidirectional biocomposites is shown in Figure 6. Composites fabricated by film-stacking method exhibited higher impact strength whereas solvent-impregnated composite showed lower impact strength. Debonding, pull out and fiber fractures are the mechanisms of energy absorption during impact. Among these, fiber pullout requires higher energy than that of the fracture of fibers and debonding. High impact strength of the film-stacked composite indicates more fiber pullouts due to weak interface between fiber and matrix, whereas lower energy absorbing capacity of solvent-impregnated composite indicates a strong fiber matrix interface due to better wettability and impregnation of fiber in the matrix. Impact strength increased with fiber content and a maximum of 58 KJ/m^2 was observed at 50% fiber weight fraction.

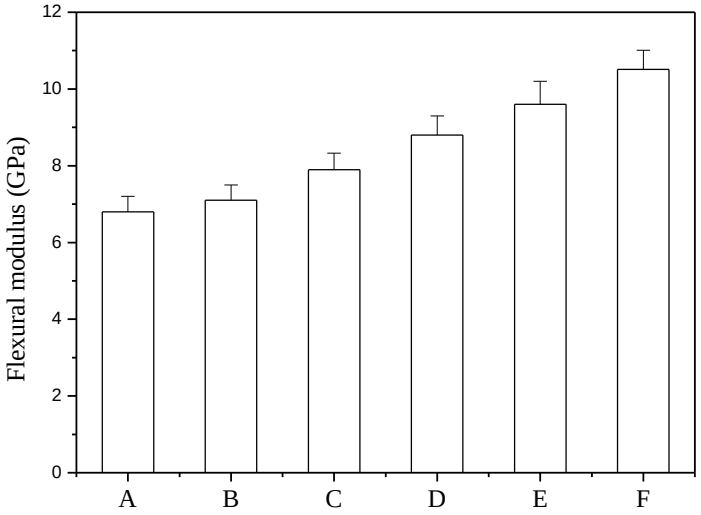

Fig. 5: *Flexural Modulus of Sisal Fiber-Reinforced Biocomposites for (A) 20 wt% Sisal Fiber FS Composite (B) 20 wt% Sisal Fiber MI Composite (C) 20 wt% Sisal Fiber SI Composite (D)30 wt% Sisal Fiber SI Composite (E) 40 wt% Sisal Fiber SI Composite (F) 50 wt% Sisal Fiber SI Composite.*

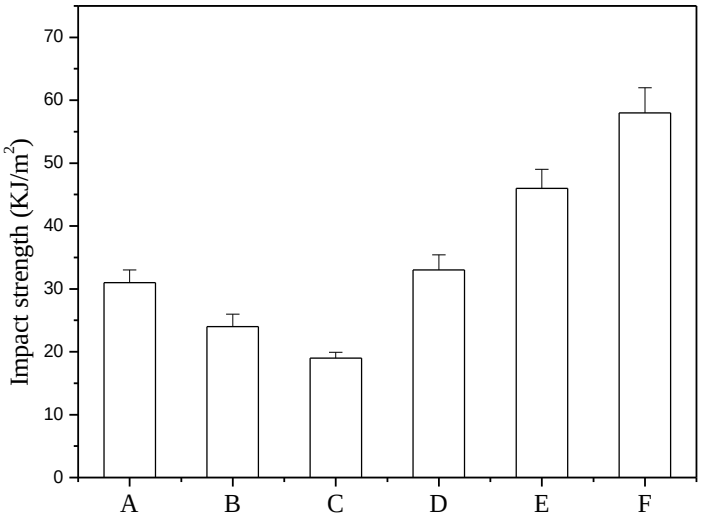

Fig. 6: *Izod Impact Strength of Sisal Fiber-Reinforced Biocomposites for (A) 20 wt% Sisal Fiber FS Composite (B) 20 wt% Sisal Fiber MI Composite (C) 20 wt% Sisal Fiber SI Composite (D) 30 wt% Sisal Fiber SI Composite (E) 40 wt% Sisal Fiber SI Composite (F) 50 wt% Sisal Fiber SI Composite.*

Dynamic Mechanical Properties

Dynamic mechanical analysis was carried out on all the composites to study the effect of processing methods and fiber content on viscoelastic behavior of unidirectional sisal fiber-reinforced biocomposites. Figure 7

shows the variation of storage modulus with temperature. Over the range of the temperatures studied, the composite fabricated with solvent-impregnation method has shown higher storage modulus when compared to other processing methods indicating a higher dynamic stiffness. Higher storage modulus of solvent-impregnated composite may be attributed to better wettability and impregnation of fiber in the matrix. In addition, the storage modulus increases with progressive increment of fiber content. Effect of processing methods and fiber content on the loss factors (tan delta) of the composites is shown in Figure 8. Among the three processing methods studied, composites fabricated with film-stacking method exhibited higher magnitude of tan δ peak, whereas solvent-impregnated composite showed lower tan δ peak. Decrease in tan δ peak value of solvent-impregnated composite may be due to strong interfacial interaction between treated fiber and matrix, which hinders the polymer chain mobility and mechanical loss to overcome inter-friction between molecular chains [16]. It has been reported earlier [17] that tan δ peak is related to impact strength of the material. As seen from Figure 8, solvent-impregnated composite gives lower magnitude of tan δ peak corresponding with its inferior impact properties, indicating the little contribution from fiber pull out mechanism. Tan δ peak decreased with the increase in fiber content. Composite with 50% fiber weight fraction has the lowest tan δ, indicating better interaction between matrix and fiber.

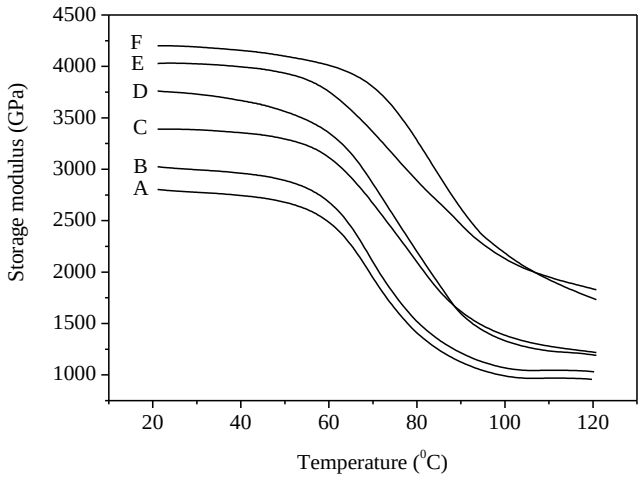

Fig. 7: *Storage Modulus versus Temperature of Sisal Fiber-Reinforced Biocomposites for (A) 20 wt% Sisal Fiber FS Composite (B) 20 wt% Sisal Fiber MI Composite (C) 20 wt% Sisal Fiber SI Composite (D) 30 wt% Sisal Fiber SI Composite (E) 40 wt% Sisal Fiber SI Composite (F) 50 wt% Sisal Fiber SI Composite.*

Abrasive Wear Performance

Abrasive wear behavior of unidirectional sisal fiber-reinforced biocomposites fabricated with different processing methods and fiber weight fractions are studied in this work. All the composites are tested in an orientation where sisal fiber mats are perpendicular to sliding distance and parallel to normal load. Figure 9 exhibits the variation of weight loss as a function of sliding distance at a constant sliding velocity 0.418 m/s. It can be observed that weight loss increased with the increasing sliding distance. During abrasion, temperature of the contact region increased with increasing sliding distance, which may loosen the bonding and hence more material removal occurred at higher sliding distances. As shown in Figure 9, the highest weight loss observed for film-stacked composite may be due to weak interfacial bonding and poor wettability between fiber and matrix. Composite fabricated with solvent-impregnation method shows better wear resistance when compared to other two processing methods. The process of wear in composites depends on the interaction between the reinforcement and matrix [13].

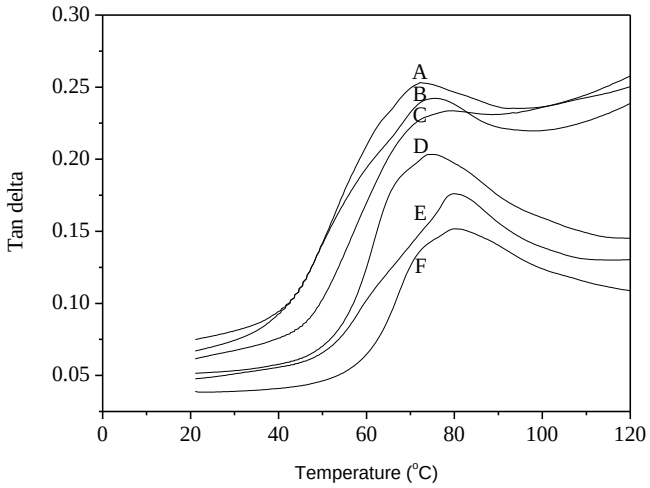

Fig. 8: *Tan Delta versus Temperature of Sisal Fiber-Reinforced Biocomposites for (A) 20 wt% Sisal Fiber FS Composite (B) 20 wt% Sisal Fiber MI Composite (C) 20 wt% Sisal Fiber SI Composite (D) 30 wt% Sisal Fiber SI Composite (E) 40 wt% Sisal Fiber SI Composite (F) 50 wt% Sisal Fiber SI Composite.*

Thus, the higher wear resistance of the solvent-impregnated composite indicates the better interaction between fiber and matrix. In addition, fiber content also influenced the abrasive wear performance of sisal fiber reinforced biocomposites. It is observed that wear resistance increased with increasing fiber content. In actual, wear resistance of fiber-reinforced composites is a complex phenomenon that depends on a number of factors such as tensile properties, toughness, hardness, fiber matrix adhesion, etc. [18]. Higher wear resistance of the composite with higher fiber weight fraction may be attributed to increased stiffness.

Morphologies of the worn surfaces of film-stacked and solvent-impregnated composites are shown in Figure 10. In all, the biocomposites removal of material is due to micro-cutting mechanism. In case of film-stacked composite, deep ploughed grooves are observed on the worn surface (Figure 10A) whereas in the solvent-impregnated composites, shallow ploughed groves or tiny scratches are observed which is a characteristic of excellent wear behavior.

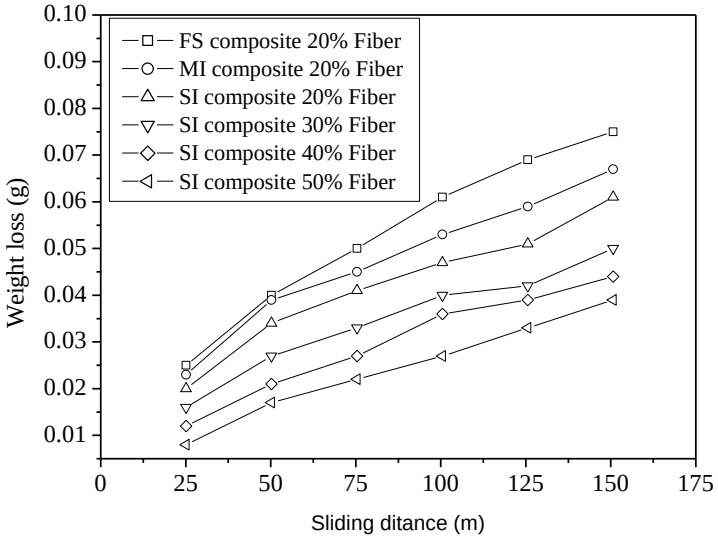

Fig. 9: *Plot between Weight Loss and Sliding Distance.*

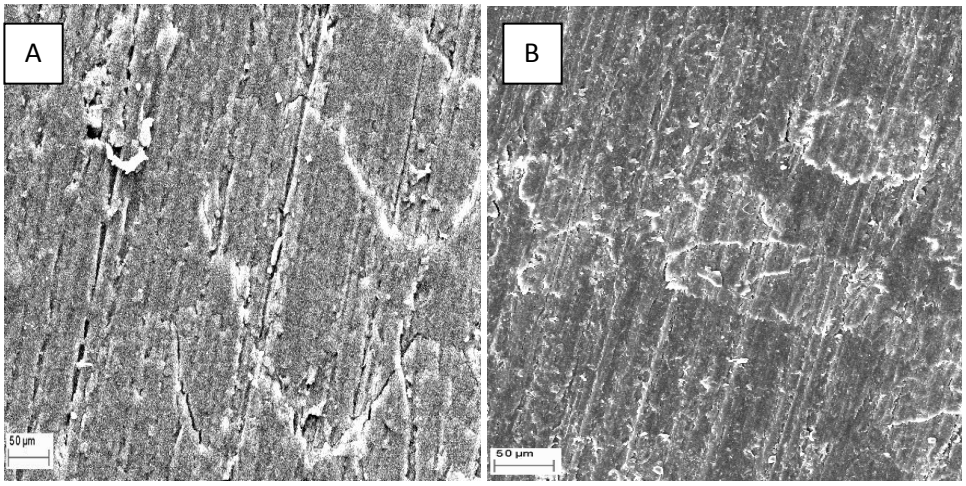

Fig. 10: *Morphologies of the Worn Surfaces of (A) 20 wt% Sisal Fiber FS Composite, (B) 20 wt% Sisal Fiber SI Composite.*

CONCLUSIONS

The objective of this study is to investigate the effect of processing methods and fiber content on mechanical, dynamic mechanical and abrasive wear behavior of unidirectional sisal fiber-reinforced composites. Solvent-impregnated composite had higher tensile and flexural properties and lower Izod impact strength when compared to composites processed by film-stacking and melt-impregnation methods. In addition, processing methods also influenced the abrasive wear performance of composites. Solvent-impregnated composite with better fiber matrix adhesion exhibited maximum abrasive wear resistance. The mechanical, dynamic stiffness and abrasive wear resistance of sisal fiber-reinforced biocomposites processed by solvent-impregnation method increased with increasing fiber content.

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